

補助事業番号 2017M-117

補助事業名 平成29年度 高繰り返しナノ秒パルスプラズマを用いたダイヤモンド
ライクカーボン成膜技術の開発 補助事業

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1 研究の概要

ダイヤモンドライクカーボン(Diamond-like carbon, DLC)はその優れた特性から機械部品のコーティング等、様々な産業分野に使われているが、低コスト化が強く望まれている。本事業では、近年開発されたSiC-MOSFETインバータ電源を用いて準大気圧高繰り返しナノ秒パルスグロー放電を生成し、DLCの高速成膜技術を開発した。特に高繰り返し周波数下のパルスグロー放電特性とDLCの膜質向上の関係を明らかにした。

2 研究の目的と背景

従来のDLC成膜技術は高真空機器が必要な低ガス圧プラズマ装置を用いており、装置コストが高く、また成膜時間が長いことが課題であった。本研究では、真空排気系としてロータリーポンプのみを用いる準大気圧プラズマプロセスにより高速DLC成膜技術を開発することを目的とする。特に最新のSiC-MOSFETインバータ電源を用いた高繰り返しナノ秒パルスグロー放電の生成とその特性解明に基づいたDLC成膜技術を開発する。

3 研究内容

(1)高繰り返しナノ秒パルスグロー放電の特性評価

<http://www.eng.u-hyogo.ac.jp/faculty/ykikuchi/JKA-ring-ring.html>

真空容器内に平行平板電極を設置し、ロータリーポンプで真空排気した後、放電ガス(ヘリウム、He)を流して圧力を10 kPaとした。SiC-MOSFETインバータ電源により高繰り返しナノ秒パルス電圧を電極間に印加し、グロー放電を生成した。また、整流器を用いることで両極性パルス電圧を負極性パルス電圧とし、周波数を30~600 kHzとした。低繰り返し周波数の30 kHzでは、電極間全体にパルスグロー放電が生成され、パルス電圧オフ後のアフターグロー発光は6 μ s程度続いた。一方、高繰り返し周波数の600 kHzでは、陰極近傍にHe原子発光線の発光が局在化し、アフターグロー発光も陰極表面にのみ存在した。このように、高繰り返しパルス電圧を印加することで、アフターグローが残存する間に次のパルス電圧を印加できることとなり、それが陰極局在化グロー放電モードを駆動することが明らかとなった。

(2)高繰り返しナノ秒パルスグロー放電を用いた高速DLC成膜

<http://www.eng.u-hyogo.ac.jp/faculty/ykikuchi/JKA-ring-ring.html>

放電ガスにHeとメタン(CH₄)の混合ガスを用いて、高繰り返しナノ秒パルスグロー放電を生成し、繰り返し周波数30~200 kHzと変化させて、シリコン基板上にカーボン膜を成膜した。30 kHzでは、カーボン膜の硬度は4 GPaと低く、周波数を高くすると硬度も上昇した。その結果、200 kHzでは硬

度13 GPaが得られた。また、ラマン分光計測の結果、sp²とsp³に由来する2つのピークが確認され、DLCの典型的なラマンスペクトルが得られた。ラマンスペクトルおよびグロー放電発光分光法から膜中の水素量を定性的に評価すると、高繰り返し周波数にすることで水素量が低下していることが分かった。

4 本研究が実社会にどう活かされるかー展望

高繰り返しナノ秒パルスグロー放電を用いることで、従来の5倍程度の成膜速度100 nm/minにて、硬度13 GPaのDLC膜が得られた。また、SiC-MOSFETインバータ電源を用いた新しいプラズマ源を提示することができ、今後様々な材料表面改質に適用が可能と考えられる。

5 教歴・研究歴の流れにおける今回研究の位置づけ

これまで、大気圧・準大気圧繰り返し放電を用いたDLC成膜技術開発を行ってきた。本事業により、従来のSi-IGBTインバータ電源に代わり、最新のSiC-MOSFETインバータ電源をプラズマ生成用電源に投入することが可能となった。本電源を用いて高繰り返しナノ秒パルスグロー放電の特性評価を行い、学術的な成果も多く得られた。世界的に未開拓な新しいプラズマ源であり、今後もこの流れを継続し、新しい研究成果を発信していく。

6 本研究にかかわる知財・発表論文等

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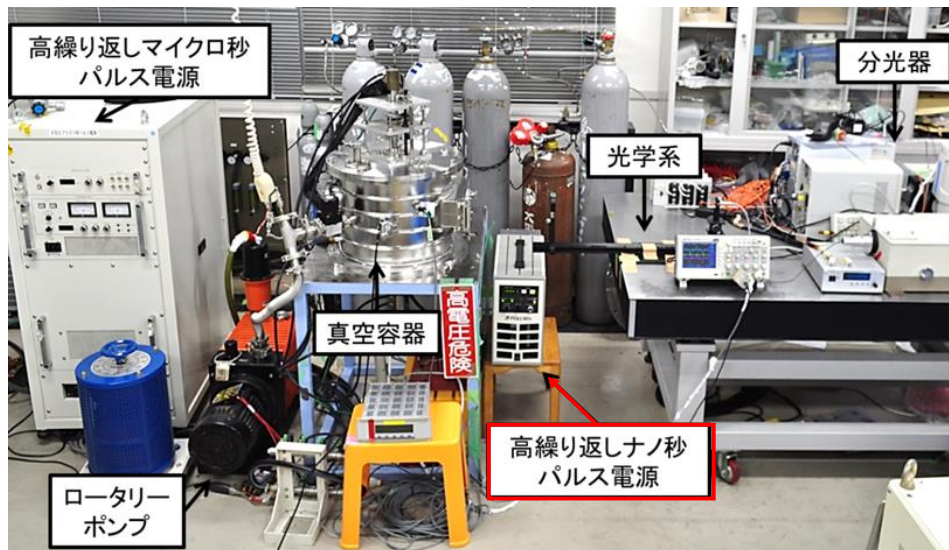
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7 補助事業に係る成果物

(1)補助事業により作成したもの

<http://www.eng.u-hyogo.ac.jp/faculty/ykikuchi/JKA-ring-ring.html>



準大気圧高繰り返しナノ秒パルスグロー放電生成装置



Diamond-like carbon film preparation using a high-repetition nanosecond pulsed glow discharge plasma at gas pressure of 1 kPa generated by a SiC-MOSFET inverter power supply

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A high-repetition nanosecond pulsed glow discharge plasma at a gas pressure of 1 kPa was generated using a SiC-MOSFET inverter power supply for diamond-like carbon (DLC) film preparation. At a high repetition frequency above 50 kHz, the period of the nanosecond voltage pulse becomes shorter than the decay time of the afterglow plasma, and many ions and radicals remained in the glow space. The deposition rate was 0.1 μm/h, which was 5 times higher than that of a conventional plasma CVD process. An increase in hardness to 13 GPa and a decrease in hydrogen content in the DLC film were confirmed by increasing the repetition frequency to 250 kHz. © 2017 The Japan Society of Applied Physics

Diamond-like carbon (DLC) films have attracted considerable attention because of their significant potential applications in a wide range of fields related to mechanical sliding parts,^{1)–3)} biomedical,^{4)–6)} and gas-barrier-improved polyethylene terephthalate bottles.⁷⁾ Currently, DLC films have been prepared by physical vapor deposition (PVD)⁸⁾ and chemical vapor deposition (CVD)⁹⁾ under low gas pressures, typically below 10 Pa. The conventional plasma-assisted CVD process resulted in the low deposition rate of DLC films owing to the use of a relatively low-density plasma and a requirement of costly vacuum systems such as a turbo molecular pump. Thus, a high-rate deposition technique without the use of costly vacuum systems is highly required in order to produce DLC films in new industrial fields.

There have so far been several studies on DLC film preparation using plasma CVD processes under sub-atmospheric/ambient pressure in order to realize a high-rate deposition technique for DLC films and cost reduction by removing the associated vacuum systems.^{10)–12)} However, arcing is classified as a harmful aspect of a sub-atmospheric/ambient pressure plasma, which would inhibit the uniform deposition of DLC films. Dielectric barrier discharge (DBD) plasmas can produce a stable sub-atmospheric/ambient pressure glow discharge plasma, but soft amorphous carbon films with a hardness of ~4 GPa have been prepared with DBD plasma processes. On the other hand, a nanosecond pulsed power supply has been used for plasma production at a sub-atmospheric/ambient pressure, because the applied voltage pulse is turned off before a glow-to-arc transition event. The deposition of DLC films using repetitive nanosecond pulsed plasmas with a pulse duration of ~100 ns and a repetition frequency of ~1 kHz under a sub-atmospheric/ambient pressure has been reported.¹³⁾ A nanopulse generator with a static induction thyristor and an inductive energy storage circuit for plasma production was used. In these experiments, however, streamer discharges were used for DLC film preparation. From an industrial viewpoint, the process with the use of streamer discharges is not appropriate for DLC film preparation because it causes the insufficient uniformity and poor adhesion of DLC films. It is expected that DLC film preparation with a stable glow discharge will overcome the disadvantages encountered by the method with streamer discharges.

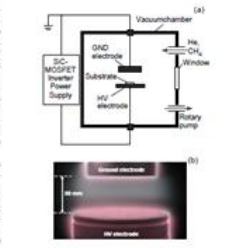


Fig. 1. (Color online) (a) Schematic view of experimental device and (b) image of a typical discharge plasma taken by a digital camera with an exposure time of 1 s.

In recent years, silicon-carbide (SiC)^{14)–16)} metal-oxide-semiconductor field-effect transistors (MOSFETs)¹⁷⁾ have been developed and have emerged in the commercial market. SiC MOSFETs have a much lower switching loss than conventional Si-based switching devices, leading to energy efficiency improvement in an inverter-driven electrical apparatus. It is expected that this novel technology will provide a potential power supply for advanced plasma sources, because it enables us to develop a nanopulse generator with a high-repetition frequency of several hundreds of kHz. In this paper, we firstly demonstrate the generation of a high-repetition nanosecond pulsed glow discharge plasma at a gas pressure of 1 kPa using a SiC-MOSFET inverter power supply and its application to the preparation of a DLC film with a high-deposition rate.

Figure 1(a) shows a schematic view of the experimental device in this study. The vacuum chamber was evacuated

Letter

Two discharge modes of a repetitive nanosecond pulsed helium glow discharge under sub-atmospheric pressure in the repetition frequency range of 20 to 600 kHz

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Abstract

Two discharge modes, α and γ , of a repetitive nanosecond pulsed helium glow discharge at a gas pressure of 10 kPa in the repetition frequency range from 20 to 600 kHz are reported for the first time. The pulsed glow discharge is produced in a pair of parallel plate metal electrodes without insertion of dielectrics. The α mode discharge is volumetrically produced in the electrode gap at a low-repetition frequency, whereas the γ mode discharge is localized at the cathode surface at a high-repetition frequency. At high-repetition frequency, the time interval between voltage pulses is shorter than the lifetime of the afterglow produced by the preceding discharge. Thus, the γ mode discharge is maintained by a large number of secondary electrons emitted from the cathode exposed to high-density ions and metastable helium atoms in the afterglow. In the α mode discharge with a low-repetition frequency operation, primary electrons due to gas ionization dominate the ionization process. Thus, a large discharge voltage is needed for the excitation of the α mode discharge. It is established that the bifurcation of α - γ discharge mode, accompanied by a decrease in the discharge voltage, occurs at the high-repetition frequency of ~120 kHz.

Keywords: pulsed glow discharge, afterglow, sub-atmospheric pressure, high-repetition frequency, discharge mode

Atmospheric and sub-atmospheric pressure discharges have been intensively studied for numerous industrial applications [1]. In particular, a glow discharge, which is spatially uniform without filaments, is suitable for material processing such as film deposition [2]. However, a high-pressure glow discharge tends to be unstable and changes into an arc discharge. It is well known that a dielectric barrier discharge (DBD) is an efficient method for the generation of a high-pressure glow discharge without a glow-to-arc transition event [3–6]. On the other hand, a high-pressure glow discharge can be transiently generated between metal electrodes without insertion of dielectrics using a nanosecond pulsed voltage application,

because the applied voltage pulse is turned off before a glow-to-arc transition event [7]. It is particularly worth noting that this technique can provide a transient glow discharge with a high instantaneous electric power consumption of several tens of kW [8].

A repetition frequency of pulsed voltage application is an important parameter in the determination of discharge characteristics. When the time interval between voltage pulses is shorter than the lifetime of an afterglow produced by a preceding pulsed discharge, charged particles and radicals in the afterglow give rise to a pre-discharge effect for a subsequent pulsed discharge. It is considered that an afterglow lifetime of

発表論文①

発表論文②

8 事業内容についての問い合わせ先

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